Carbon Nanotube Material Quality Assessment

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Abstract

The nanomaterial activities at NASA-Johnson Space Center focus on carbon nanotube production, characterization and their applications for aerospace systems. Single wall carbon nanotubes are produced by arc and laser methods. Characterization of the nanotube material is performed using the NASA-JSC protocol developed by combining analytical techniques of SEM, TEM, UV-VIS-NIR absorption, Raman, and TGA [1]. A possible addition of other techniques such as XPS, and ICP to the existing protocol will be discussed. Changes in the quality of the material collected in different regions of the arc and laser production chambers is assessed using the original JSC protocol. The observed variations indicate different growth conditions in different regions of the production chambers.

Ref.: 1) Arepalli S., Nikolaev P., Gorelik O., Hadjiev V. G., Holmes W. A., Files B. S., and Yowell L., "Protocol for the Characterization of Single-Wall Carbon Nanotube Material Quality", Carbon, Vol. 42, pp. 1783-1791 (2004).

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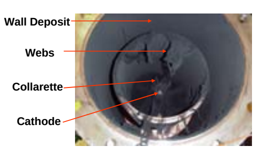
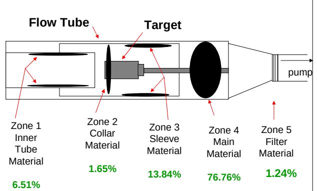
NASA-JSC Protocol for SWCNT Characterization

- **Thermogravimetric Analysis (TGA), (TA SDT 2960)**
  3 runs using 3-4 mg of material in100 sccm air at a 5 ºC/min heating rate from room temperature to 1000˚C.
- **Transmission Electron Microscopy (TEM) & Energy Dispersive X-ray Spectroscopy (EDS), (JEOL 2010 FX)**
  Small quantity dispersed in methanol, sonicated then place on TEM grid
- **Scanning Electron Microscopy (SEM) & EDS (Phillips XL40 FEG)**
  Image 3 locations at 20 and 50kX with corresponding EDS spectra at same locations
- **Raman Spectroscopy (Renishaw RM 1000, Horiba Yvon Jobin)**
  Spectra collected in three different locations with 780, 633 and 514 nm excitation sources
- **UV-Visible-NIR spectrometry (Perkin-Elmer Lambda 900)**
  0.1 mg sample placed in 10 mL Dimethyl Formamide (DMF) and sonicated until well dispersed

Production and Collection

Arc Discharge Method | **Pulsed Laser Vaporization**





Arc Production Conditions:

3.92%Ni:1%Y, Pressure:506 Torr, Voltage:38.2V, Current:101.5A, Electrode Distance:3 mm, Automated

Laser Production Conditions:

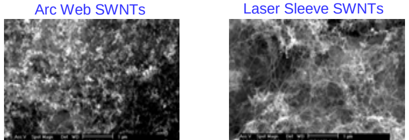
3/4" diameter target, 1%Co:1%Ni, Pressure: 500T, Ar Flow Rate: 100 sccm, Pulse Separation: 50 ns , Power Density: 1.6J/cm^2, Oven Temperature: 1200 ºC, Laser Sequence: Green-IR

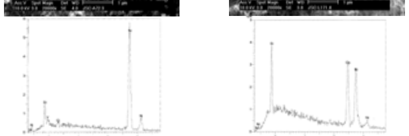
JSC Characterization Protocol for SEM

SEM Analysis

1. Images: Qualitative information about **impurities, general morphology of the sample and its homogeneity**
2. EDS: Qualitative information about about **metals, silicon and chlorine impurities**

Arc Web SWNTs | Laser Sleeve SWNTs





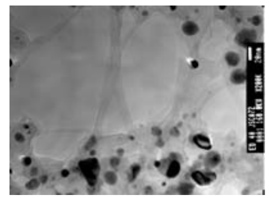
SEM images of other locations show very little difference in the morphology of the various materials.
EDS can not provide any indication on the variation of non-carbon impurity levels or their chemical states.

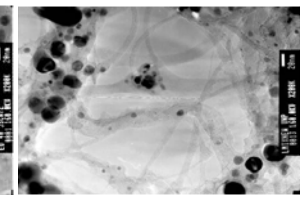
JSC Characterization Protocol for TEM

TEM Analysis

1. Images: Qualitative information about **non-nanotube carbon impurities** ("schmutz" and graphitic particles) and their distribution within a sample
2. Images: Qualitative information about **metal content**

Arc SWNT Material | Laser SWCNT Material



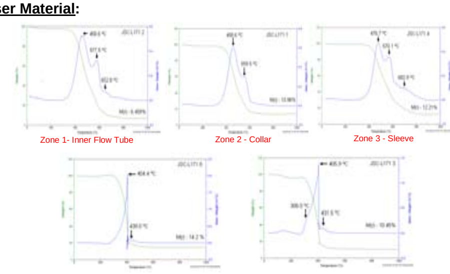


JSC characterization Protocol for TGA

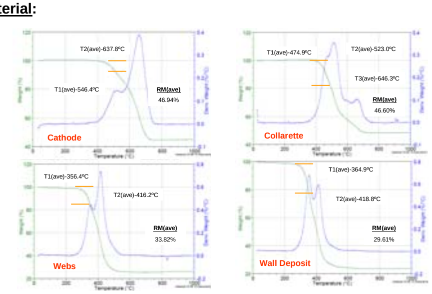
Information extracted from TGA data:

1. Average residual mass M_r (in %): Shows **fraction of residual metals** in the specimen.
2. Temperature T_m of the maximum in the burning rate dm/dT : Shows **thermal stability** of the specimen.
3. Standard deviation of M_r and T_m: Shows **homogeneity** of the specimen

Laser Material:



Arc Material:



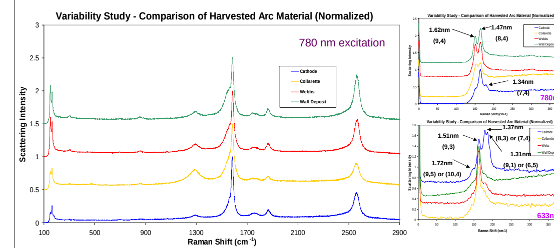
Additional Techniques

- **ICP can provide a better quantitative measure of of the metal impurity levels with as produced and purified materials (digestion of metals major issue).**
- **XPS can provide information on the chemical states of non-carbon impurities to assist with TGA and ICP analysis.**

JSC Characterization Protocol for Raman Spectroscopy

Analysis of Raman Spectra

1. Nanotube protonation state from the C-C stretch mode shift.
2. Possible information about impurities and disorder in the sample from the 1340 cm^{-1} disorder peak position and width
3. Qualitative information about sample homogeneity from the variability in the spectra

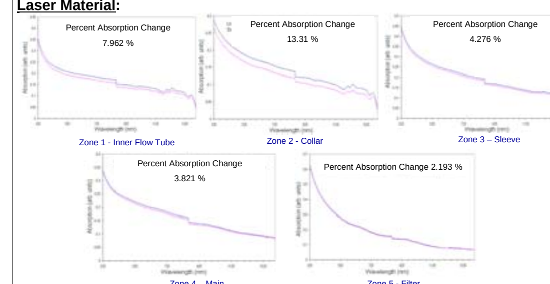


JSC Characterization Protocol for UV-Visible-NIR Spectroscopy

Analysis of UV-Vis-NIR Spectra

- Provides quantitative information about **how well nanotubes stay in suspension**
- Can provide information on electronic property of tubes (metallic, semiconductive)
- Possible determination of the purity of nanotubes

Laser Material:



Analysis Results Summary Table for Laser Material

PROPERTIES	MATERIAL: Inner	Collar	Sleeve	Main	Filter
Residual Mass	6.45%	13.98%	12.21%	14.26%	10.45%
Thermal Stability	Min 458.6 ºC Max 652.8 ºC	Min 458.6 º C Max 559.5 º C	Min 476.7 º C Max 682.9 º C	Min 404.4 º C Max 439.0 º C	Min 405.9 º C Max 431.5 º C
Dispersion	7.962%	13.31%	4.276%	3.821%	2.193%
D/G Ratios	0.288	0.090	0.047	0.094	0.124
D-Band Position	1285.45cm^{-1}	1289.9cm^{-1}	1287.87cm^{-1}	1284.84cm^{-1}	1283.17cm^{-1}
Small Diameter % Raman	8.02%	22.6%	8.17%	9.85%	27.5%